

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
28 December 2000 (28.12.2000)

PCT

(10) International Publication Number
WO 00/79616 A1

(51) International Patent Classification: H01L 51/20

(72) Inventors; and

(21) International Application Number: PCT/GB00/02377

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(22) International Filing Date: 19 June 2000 (19.06.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
9914372.9 18 June 1999 (18.06.1999) GB
9927116.5 16 November 1999 (16.11.1999) GB

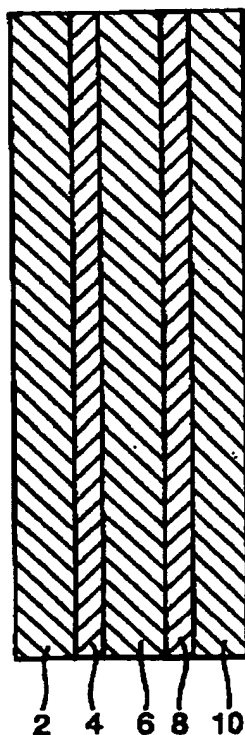
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(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ,

[Continued on next page]

(54) Title: FLAT PANEL DISPLAY WITH IMPROVED CONTRAST



(57) Abstract: A light emitting device is described which comprises a transparent substrate layer (2), a transparent electrode layer (4), a light emitting layer (6) and a back electrode (10) which is in the form of a layer of carbon.

WO 00/79616 A1



NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM,
TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

Published:

— With international search report.

- (84) **Designated States (regional):** ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

FLAT PANEL DISPLAY WITH IMPROVED CONTRAST

5 This invention relates to a non-reflective electrode which can be used in a flat panel display.

Flat panel displays are the critical enabling technology for many current applications including laptop computers, portable displays and "head up" displays. 10 Currently the flat panel display market is dominated by liquid crystal technology, but these materials are non-emissive requiring the light source to be filtered which reduces the efficiency of the device. Much work is currently being undertaken on developing alternative 15 emissive technologies including organic electroluminescent (EL) displays.

One of the factors affecting the viewing quality of a display is the contrast. Contrast is the difference in 20 brightness between when a pixel is on and when it is off. In practice, a proportion of the ambient light that is incident on a device is reflected, so that the total light observed is the sum of the emitted light and the reflected ambient light. If the proportion of reflected 25 ambient light is relatively large, then the contrast is poor. This is frequently the case with the current display devices. There is therefore a need to find a way of increasing contrast.

30 According to the present invention there is provided a light emitting device which comprises sequentially a transparent substrate layer, a transparent electrode layer, a light emitting layer and a back electrode which is in the form of a layer of carbon and an at least semi- 35 transparent intermediate layer between the light emitting

layer and the back electrode, said intermediate layer having its lowest empty energy level or levels, or lowest empty energy band, positioned above the conduction band of the carbon layer when the two layers are brought into
5 contact. In current organic EL devices the back electrode is a metal such as aluminium which is highly reflective and consequently the contrast is poor. By replacing this metal by a carbon layer which is substantially black contrast can be improved significantly.

10

In order that the energy gap difference between the light emitting layer and the back electrode should be at an appropriate level it will be appreciated that the nature of the light emitting layer should be selected
15 appropriately. The intermediate layer is at least semi-transparent ie. it should be at least 50% transmissive, preferably at least 70% transmissive and more preferably at least 90% transmissive in the visible region (380 to 780 nm). This intermediate layer serves to improve
20 electron injection into the device, possibly by lowering the barrier to injection and/or improving the conductivity of the electrode. Without such a layer it is generally found that it is necessary to operate at a higher voltage since the carbon layer does not function
25 so effectively as a source of electrons. This semi-transparent intermediate layer may be defined as the layer having its lowest empty energy level or levels, or lowest empty energy band, positioned above the conduction band of the carbon layer when the two layers are brought
30 into contact.

The intermediate layer is generally made of a semi-transparent metal or alloy or is a dielectric layer. Metals with a lower work function than carbon generally
35 have empty energy levels above the conduction band of

carbon. Dielectric materials generally have a large energy separation between the full HOMO (or valence band) and empty LUMO (or conduction band) and to be effective the LUMO would have to be positioned above the carbon conduction band. It will be appreciated that the values for most metals and dielectrics are known or can, if necessary, be determined. The intermediate layer is advantageously made of a low work function metal or alloy such as magnesium, which is preferred, aluminium, silver, calcium, lithium and indium or an alloy such as Mg/Ag or Mg/Al and Li/Al. Alternatively, the semi-transparent layer may be of a dielectric material generally of a solid ionic metal or metalloid salt, typically an oxide or halide, for example a chloride or fluoride, of a metal, typically of Group I, II or III, especially IA, IIA, or IIIA, of the Periodic Table (see Advanced Inorganic Chemistry, 5th edition, Cotton v Wilkinson) such as lithium, magnesium or sodium. Specific examples include lithium chloride and fluoride, lithium oxide, magnesium oxide, magnesium fluoride, sodium chloride, caesium fluoride and chloride, barium chloride and fluoride, silicon dioxide and aluminium oxide. Desirably the dielectric has a band gap of at least 4.5 electron volts.

25

Although metal films are normally highly reflective, a sufficiently thin metal layer is semi-transparent, transmitting rather than reflecting a large proportion of the incident visible light. When a carbon layer is deposited behind such a thin metal layer, a significant proportion of the incident light is ultimately absorbed by the carbon film rather than reflected.

30

The thickness of the intermediate layer will depend to some extent on its nature. Generally, a metal or

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alloy layer will have a thickness from 500 to 0.5 nm, typically 50 to 1 nm, and especially from 25 to 2 nm. Generally if the intermediate layer is made of a dielectric material, lower thicknesses are desirable, a
5 general range being from 100 to 0.1 nm, typically 10 to 0.5 nm and preferably from 5 to 1 nm; it is preferably less than 10 nm.

It will be appreciated that all the layers in the
10 device should generally be as uniform and as continuous as possible. Obviously the continuity of the layer becomes more difficult as its thickness is reduced below certain levels. Indeed with thicknesses of the order of 2nm or less it may not be possible to form a continuous
15 layer; rather "islands" of material are present. These can still be effective.

The source of carbon used for the carbon layer is not particularly critical and includes isotropic
20 graphite, anisotropic graphite, vitreous carbon and carbon fibre although isotropic graphite of high purity is generally considered to be the best. Generally, the carbon layer will be thicker than the intermediate layer. The general thickness will range from 5 μ m to 5 nm,
25 typically from 5 μ m to 500 nm if the layer is produced by, for example, screen printing or spraying. On the other hand, if the layer is produced by, for example, sputtering, evaporation or low temperature chemical deposition thinner layers will generally be more
30 suitable, for example, from 200 to 5 nm and especially from 60 to 10 nm. The precise nature of the deposition is unimportant provided that the layer is as uniform as possible and continuous and that the carbon layer absorbs generally at least 10% of the visible light and
35 preferably at least 25%, especially at least 50%, of the

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visible light.

Other layers can be incorporated into the device of the present invention, typically charge transport layers
5 between the light emitting layer and one or both electrodes. In addition, in one embodiment, a metal or alloy layer is placed behind the back electrode. This serves several purposes. It acts to protect the back
10 electrode and it generally will have high electrical conductivity. Also the presence of the metal layer can assist production. This is because carbon deposition is relatively slow. Once the carbon layer has built up to a sufficient thickness to enhance contrast it is then
15 beneficial to switch to the more rapid metal deposition. Apart from encapsulating the carbon layer it also serves to conduct heat away from the device which may increase the stability and life of the device. In general the metal layer, which can be of the same metals or alloys as
20 discussed above in relation to the intermediate layer, for example aluminium, will be significantly thicker than the immediate layer, typically from 10 to 2000 nm. The metal layer is generally of a comparable thickness to the carbon layer. Thicker layers can be attached to the back electrode in the form of foils. Any good heat conducting
25 metal can be used for this purpose although, naturally, a relatively light metal is preferred in order to reduce the overall weight of the device. Typically aluminium foil can be used for this purpose. Such a foil acts as a heat sink which facilitates high brightness where higher
30 operating powers increase the heat dissipated in the device. Such layers are typically attached to the back electrode by adhesion, for example using an epoxy resin. For ease of manufacture, the epoxy resin is preferably one which is curable at room temperature. Also it is
35 preferred that the resin should not release byproducts on

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curing which could adversely affect the other components of the device. Accordingly, it is desirable that the epoxy resin should be one which is catalytically curable while, of course, it should be thermally conducting.

- 5 Typical examples of suitable epoxy resins which can be used include Loctite 315 and the thermally conductive 199-1402 epoxy resins from RS components.

10 In a typical embodiment of the present invention, the transparent electrode is the anode and the rear electrode forms the cathode. An electron injecting and/or transporting layer is often advantageously inserted between the light emitting layer and the cathode. Similarly a hole injecting and/or transporting
15 layer may often advantageously be inserted between the light emitting layer and the anode. The nature of such layers is known in the art. Typical materials which can be used for this purpose include polyphilic compounds, aromatic tertiary amines including compounds in which the
20 tertiary nitrogen atom is attached to 3 phenyl rings such as N,N'-diphenyl-N,N'-bis-(3-methyl)-1,1'-biphenyl-4,4'-diamine(TPD), stilbenes, triazoles, oxadiazoles such as BBO (2-(4-phenyl)-5-(4-t'-butylphenyl)-1,3,4-oxadiazole, imidazoles. Other suitable materials can be
25 found in US Patent No. 5,756,224 and Macromol. Symp. 125, 1-48 (1997)

The nature of the light emitting layer is not particularly critical although, as indicated above, it
30 should be selected such that there is a suitable energy gap between it and the carbon layer. Suitable materials include organic and organo metallic molecular species including organolanthanide complexes such as di-or trivalent lanthanide metal ions complexed with one or
35 more polydentate ligands containing one or more

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pyrazolyl-derived groups e.g. trispyrazolyl borates anions (see GB Application No. 9820805.1 and WO 98/55561), light emitting polymers for example carbazoles such as PVK (poly-(9-vinyl carbazole) and quinolate complexes such as ALQ (tris-(8-hydroxy-quinoline) aluminium. Further details can be found in EP - 0120673 and US Patent Nos. 5,756,224 and 5,792,567.

It is frequently advantageous also to include layers which promote adhesion between the light emitting layer and the transport layers which can be present on one or both sides of it.

In general taking the light emitting layer with its associated adhesion and transport layers, the total thickness will be from 10 nm to 1 micron and especially from 60 to 150 nm with the transport layers generally having a thickness from 20 to 80 nm.

The transparent electrode which typically forms the anode of the device is preferably made from indium tin oxide (ITO) although other similar materials including indium oxide/tin oxide, tin oxide/antimony and zinc oxide/aluminium can also be used. Conducting polymers such as PANI (polyaniline) and PEDT may also be used.

The present invention also provides a process for preparing the device which comprises depositing a transparent electrode on a transparent substrate, sequentially applying over the transparent electrode, the light emitting layer and the back electrode.

The present invention will now be illustrated, merely by way of example, with reference to the accompanying Figures in which:

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Figure 1 shows, schematically, a typical preferred light emitting device of the present invention.

5 Figure 2 shows the current-voltage characteristics of a device of the present invention and of comparative devices.

10 Figure 3 shows an electroluminescent spectrum of a device of the present invention and,

Figure 4 shows the cathode reflectivity of a device of the present invention compared with a comparative device.

15 Referring to Figure 1, a light emitting device is shown which comprises a transparent substrate 2, a transparent front electrode 4, a light emitting layer 6, an intermediate layer 8, and a carbon back layer 10. An eye 1 indicates the front (viewing side of the device).

20 The transparent substrate 2 is typically made of glass although other transparent dimensionally stable materials such as polyesters including PET, acrylic resins and polyamides such as nylon can also be used.

25 It will be appreciated that the devices are typically made pixelated or patterned. Thus in one embodiment the transparent electrode can be formed using standard lithography to provide electrodes as little as, for example, 10 x 10 microns. Alternatively the carbon back electrode is deposited, for example, through a shadow mask, to give an array of contacts which may be as little as, say, 20 nm. Alternatively, both electrodes are made pixelated.

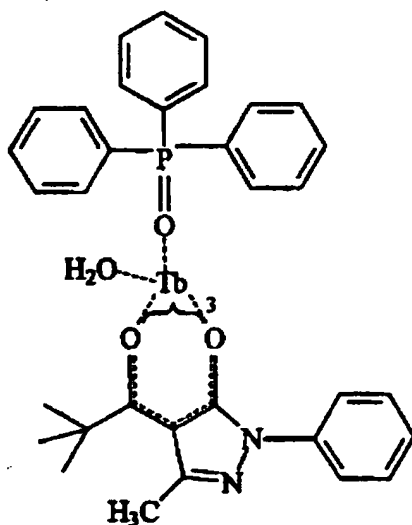
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The electrode which is not pixelated can be as large as desired, typically for a laptop computer with a diagonal dimension of 17 inches.

5 The present invention will now be further illustrated by the following Examples in which the following abbreviations are used:-

10 TPD - N,N'-diphenyl-N,N'-bis(3-methyl)
1,1'-biphenyl-4,4'-diamine. METB013 complex - a
terbium complex having the formula



TAZ - 3-(4-biphenyl)-4-phenyl-5-(4-tert-butylphenyl)-1,2,4-triazole.

15

Preparation of devices and EL testing

Low resistance ITO coated glass (20 ohms/sq) was cut

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into 1" x 1" (2.5 x 2.5cm) plates and patterned by standard lithography (HCl etchant) to give continuous electrodes (0.2mm x 0.8mm). The patterned ITO plates were ultrasonically cleaned for 30 min in a boiling ammonia and hydrogen peroxide aqueous solution (RSA process), and then dried in an oven at 100°C. After cleaning and drying, the substrates were placed inside a vacuum evaporator with a base pressure better than 1×10^{-6} Torr and the organic layers were sequentially evaporated from Mo boats at a deposition rate of 0.1-0.4 nm/s as measured by a calibrated crystal thickness monitor. In all the Examples the same organic layers were used. The hole transporting layer was a 40 nm thick layer of TPD, the light emitting layer was a 34 nm thick layer of the METb013 and the electron transporting layer was a 60 nm thick layer of TAZ. After deposition of the organic layers the rear electrode(s) was deposited. The thickness and details of the contacts is explained in the individual Examples.

20

All electrical testing and optical measurements were performed under ambient conditions with no protective coating applied to the devices. EL measurements were made under forward bias (ITO positive) and the emission output was viewed in the forward direction through the transparent ITO electrode. The current-voltage (I-V) characteristics were measured with a Thurlby-Thandar TSX3510P programmable DC power supply and a Keithley 617 programmable electrometer both controlled by IBM compatible PC via IEEE488 interface. EL characteristics of the devices including the spectral and power dependencies of light output were measured with a LOT-Oriel Instaspec IV charge-coupled device (CCD) detector attached to an Oriel Multispec 1/8 M spectrograph with 400 lines/mm ruled grating. A fibre lined sighting optic

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was used to focus the image of the device to the entrance of the spectrograph. The system was calibrated with an Oriel 200 W QTH calibrated lamp connected to an Oriel 300 W radiometric power supply. Values of radiance and luminance were also measured with an International Light IL1700 Research Radiometer equipped with a calibrated Si photodetector. Transmission and reflection was measured with a Perkin-Elmer Lambda 19 spectrometer.

10 EXAMPLE 1 (Comparative)

For comparative purposes an organic electroluminescent device was produced with a conventional Al/Mg metal cathode and a patterned ITO electrode. The organic layers were deposited according to the description above and then Mg and Al were sequentially evaporated (10-20 nm Mg and 100-120 nm Al) to form the contact.

ITO/TPD 40 nm/METb013 30nm/TAZ 60 nm/Mg/Al
20 The IV curve is shown in fig 2 and the transmission curve is shown in fig 4.
At 18V, 0.4mA/cm² and 20 cd/m² the efficiency was 4.8cd/A or ~2% external quantum efficiency. The maximum brightness of the device was 77 cd/m² at 22V, and 2.7 mA/cm² which corresponds to an efficiency of 2.8 cd/A.

EXAMPLE 2

ITO/TPD 40 nm/METb013 30 nm/TAZ 60 nm/Mg 2 nm/C 20 nm/Mg/Al.
30 An organic electroluminescent device was prepared with a "black" electrode instead of an Al/Mg cathode. A 2 nm intermediate layer of Mg (evaporation rate 0.1-0.2 nm/s) followed by a 20 nm thick layer of carbon, followed by 20 nm of Mg,
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followed by ~100 nm of Al were sequentially evaporated on top of the organic layers. The carbon was deposited by evaporation at a rate of 1 nm/s and a pressure of $<1 \times 10^{-6}$ Torr. The carbon source was carbon fibre.

The IV curve is shown in Figure 2, while Figure 3 shows the EL spectrum, and Figure 4 shows the transmission curve of the device.

Table 1 lists the best efficiency and best brightness of each device.

EXAMPLE 3

ITO/TPD 40 nm/METb013 30 nm/TAZ 60 nm/C 5 nm/Mg/Al.

In this Example a 5 nm carbon film was directly deposited on to the organic layer followed by a 100 nm Al/Mg layer. A much higher voltage was required to achieve a similar brightness to that from the device in Example 2. This Example illustrates the significance of the thin metal layer under the carbon film. When thicker carbon films were deposited directly on the organic layers the device performance was poor.

The IV curve shown in Figure 2.

Table 1

Example	Cathode structure	Turn on voltage (V)	Best efficiency	Best brightness (cd/m ²)
Device 1	Mg/Al		4.8 cd/A @ 18V, 0.4 mA/cm ² , 20 cd/m ²	77 cd/m ² @ 22V, 2.7 mA/cm ² , 2.8 cd/A
Device 2	Mg (2 nm) C (20nm) Mg/Al	7 - 8.5	5.7 cd/A @ 16V, 0.16 mA/cm ² , 9.1 cd/m ²	68 cd/m ² @ 22V, 3.5 mA/cm ² , 1.9 cd/A
Device 3	C (5 nm) Mg/Al	9.5	5.2 cd/A @ 24V, 0.16 mA/cm ² , 8.3 cd/m ²	97 cd/m ² @ 33V, 5 mA/cm ² , 0.07 cd/A

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CLAIMS

1. A light emitting device which comprises sequentially a transparent substrate layer, a transparent electrode layer, a light emitting layer and a back electrode which is in the form of a layer of carbon, and an at least semi-transparent intermediate layer between the light emitting layer and the back electrode, said intermediate layer having its lowest empty energy level or levels, or lowest empty energy band, positioned above the conduction band of the carbon layer when the two layers are brought into contact.
2. A device according to claim 1 in which the intermediate layer is thinner than the back electrode layer.
3. A device according to claim 1 or 2 in which the intermediate layer is formed of magnesium, aluminium, silver, calcium, lithium or indium or an alloy thereof.
4. A device according to claim 1 or 2 in which the intermediate layer is formed of a solid oxide or halide of a metal of Group I,II or III of the Periodic Table.
5. A device according to claim 4 in which the intermediate layer is formed of lithium fluoride, lithium oxide, magnesium oxide or magnesium fluoride.
6. A device according to claim 4 or 5 in which the intermediate layer is less than 10 nm thick.
7. A device according to any one of claims 1 to 6 which also comprises a metal or metal alloy layer behind the back electrode layer.
8. A device according to any one of the preceding claims which also comprises one or more charge transport layers between the light emitting layer and the transparent electrode and/or the back electrode.
9. A device according to claim 1 substantially as hereinbefore described.
10. A process for preparing a light emitting device as

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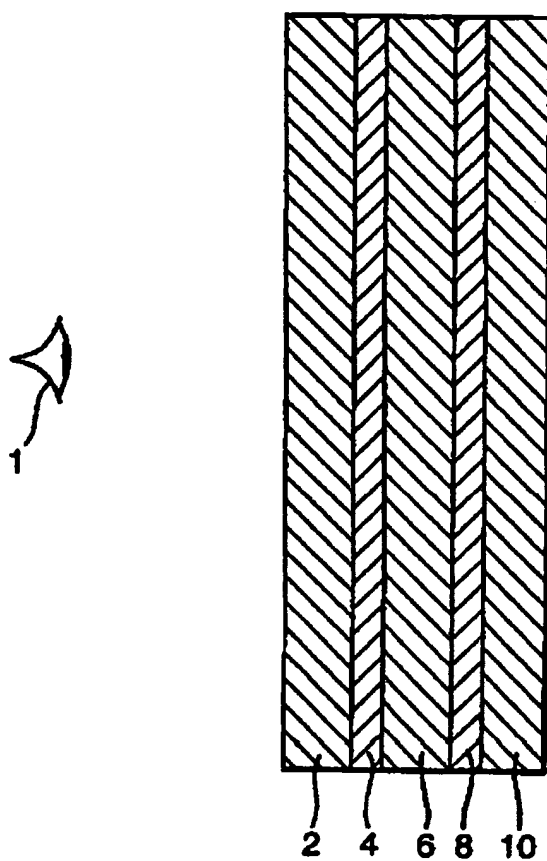
claimed in any one of the preceding claims which comprises depositing a transparent electrode on a transparent substrate, sequentially applying over the transparent electrode, the light emitting layer, the intermediate layer and the back electrode.

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11. A process according to claim 10 in which the light emitting layer and back electrode are deposited by evaporation under vacuum.

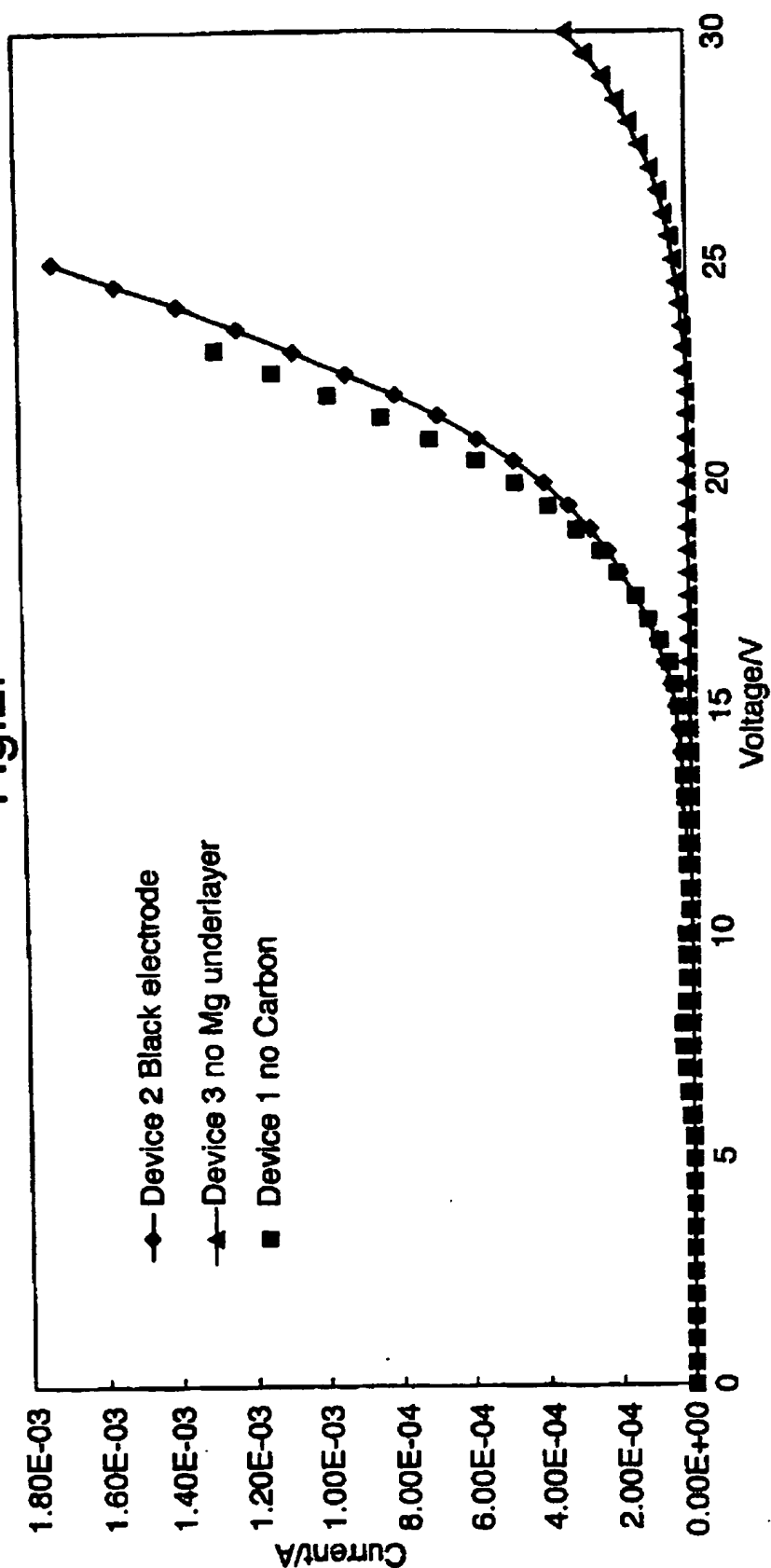
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Fig.1.

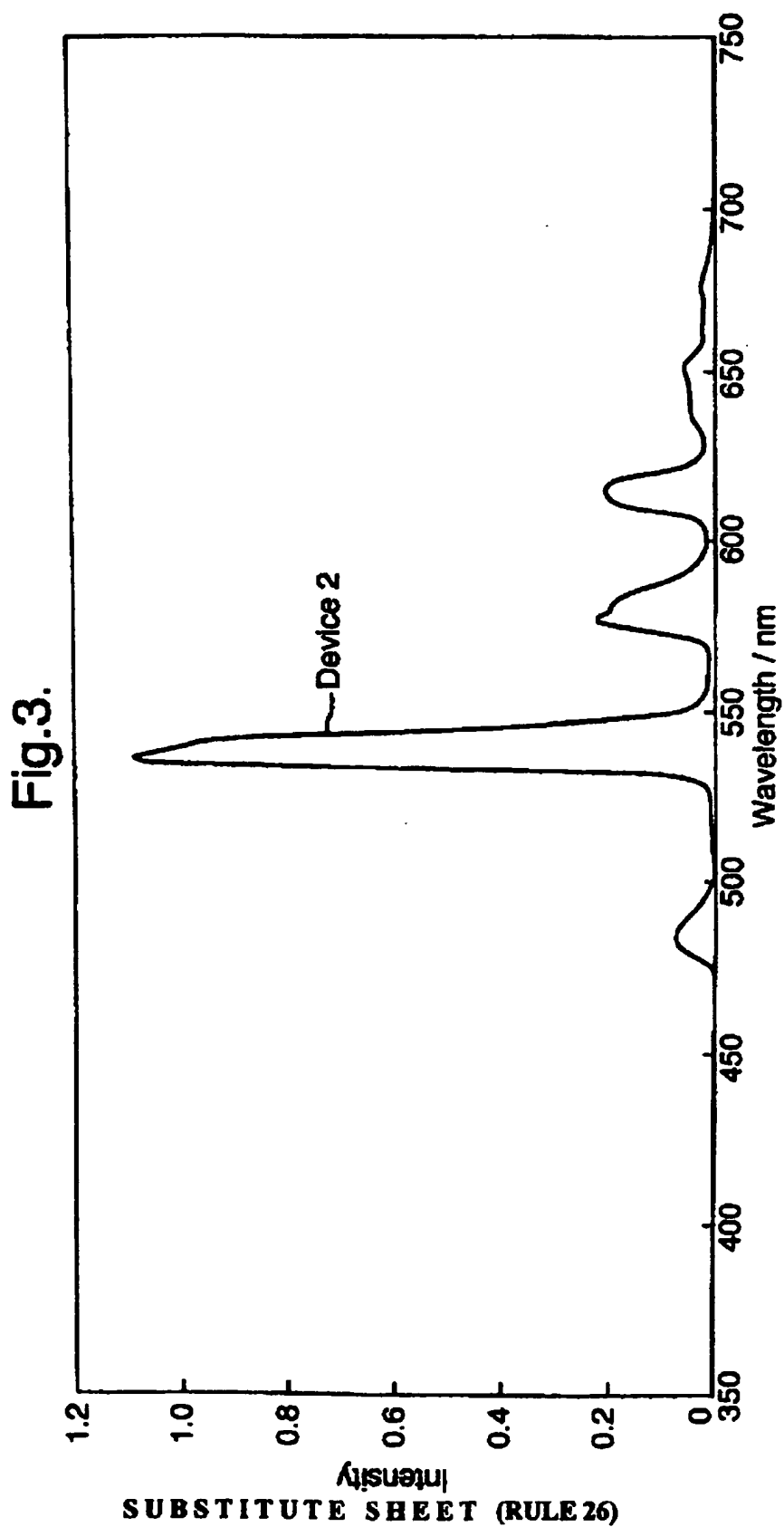


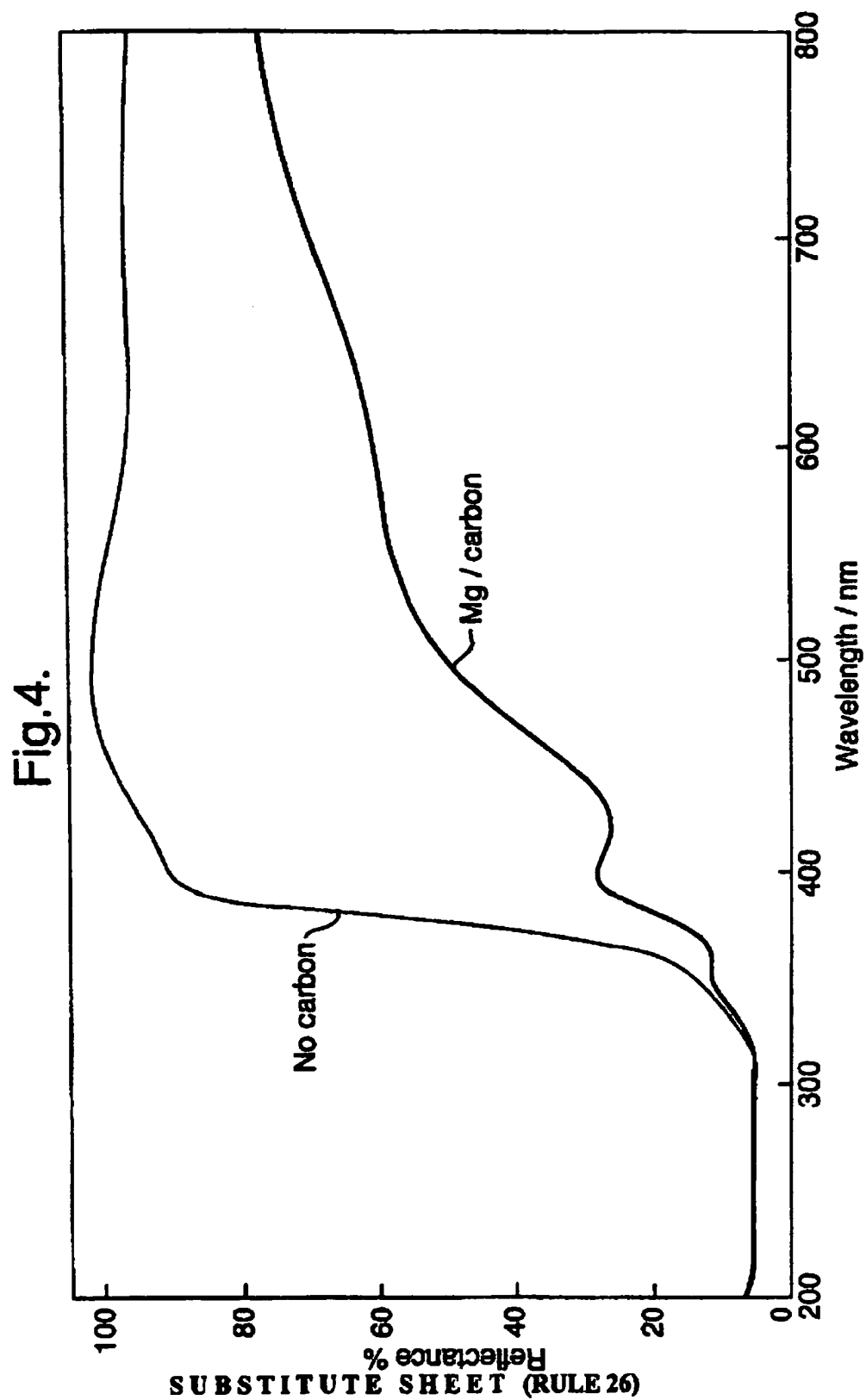
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Fig.2.



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INTERNATIONAL SEARCH REPORT

International Application No.
PCT/GB 00/02377

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H01L51/20

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 H01L H05B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO 00 35028 A (CARTER JULIAN CHARLES ;BURROUGHS JEREMY HENLEY (GB); CAMBRIDGE DI) 15 June 2000 (2000-06-15) page 8, line 13 -page 9, line 12; figure 2	1-3,6-10
A		4,5,11
Y	PATENT ABSTRACTS OF JAPAN vol. 1996, no. 05. 31 May 1996 (1996-05-31) -& JP 08 008065 A (TOPPAN PRINTING CO LTD), 12 January 1996 (1996-01-12) abstract; figure 2	1-4,6-11
	-/-	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

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Date of the actual completion of the international search

4 September 2000

Date of mailing of the international search report

13/09/2000

Name and mailing address of the ISA

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INTERNATIONAL SEARCH REPORT

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PCT/GB 00/02377

C. (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	PATENT ABSTRACTS OF JAPAN vol. 018, no. 195 (E-1533), 5 April 1994 (1994-04-05) - & JP 06 005367 A (PIONEER ELECTRON CORP), 14 January 1994 (1994-01-14) abstract; figure 4	1-4, 6-11
A	GYOUTOKU A ET AL: "AN ORGANIC ELECTROLUMINESCENT DOT-MATRIX DISPLAY USING CARBON UNDERLAYER" SYNTHETIC METALS, CH, LAUSANNE, vol. 91, no. 1/03, 21 May 1997 (1997-05-21), pages 73-75, XP000890057 ISSN: 0379-6779 abstract page 74, column 2, line 7-16	1

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Form PCT/ISA/210 (continuation of second sheet) (July 1992)

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INTERNATIONAL SEARCH REPORT

Information on patent family members

Int. Serial Application No

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